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AIR POLLUTION TESTING OF HYPERGOLIC FUEL VAPOR SCRUBBERS AT CAPE CANAVERAL AIR FORCE STATION, FLORIDA

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Hypergolic fuel and oxidizer scrubbers were studied to determine emissions produced during actual hypergol transfers. Scrubber liquor used in the fuel scrubber was 14 percent citric acid and the scrubber liquor in the oxidizer scrubber was 5 percent NaOH - 12 percent Na_2SO_3.

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The fuel (monomethylhydrazine-MMH) scrubber was studied during transfers from bulk tanks to a truck tanker. During fuel transfers the inlet and outlet MMH concentrations were monitored and the scrubber liquor was also sampled for pH and MMH concentration. Results obtained show excellent scrubber efficiency and detail scrubber performance over a course of many transfers. MMH concentrations at the inlet averaged approximately 89,000 ppm and outlet concentrations ranged from 1.1 to 2.3 ppm.

The oxidizer (Nitrogen Tetroxide - N_2O_4) scrubber was studied under similar transfer conditions. Inlet and outlet NO_2 concentrations were measured and scrubber liquor was analyzed for pH and SO_3 concentration. Average inlet NO_2 concentration for the test series was approximately 428,000 ppm and outlet concentrations ranged from approximately 1,100 to 60 ppm. Inlet and outlet N_2O_4 concentrations and scrubber liquor analyses are given for 11 transfer operations. Scrubber efficiency was generally good but capacity of the liquor was low only lasting approximately 2.5 hours under the test transfers.

PREFACE

The work described herein was conducted by Engineering Science, 7903 Westpark Drive, McLean, Virginia 22102, under U.S. Air Force Contract Number F 33615-80-D-4001. Work was performed at the Cape Canaveral Air Force Station, Florida. Major William E. Normington managed the program for the U.S. Air Force Occupational and Environmental Health Laboratory. Mr. Thomas B. Stauffer of the Air Force Engineering and Services Center supplied technical direction.

This report has been reviewed by the Public Affairs Office and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication.

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SECTION I

INTRODUCTION

BACKGROUND

The National Aeronautics and Space Administration (NASA) has several wet scrubbers designed to remove hypergolic fuel vapors from various gas streams. The scrubbers are located at Cape Canaveral Air Force Station, Florida (CCAFS). The U.S. Air Force (USAF) is interested in determining if the hypergolic scrubbers are applicable to certain USAF operations involving hypergolic fuels. Towards this end, a contract was let to conduct a series of tests at the CCAFS. The purpose of these tests was to determine the effectiveness of the scrubbers in controlling hypergolic fuel vapors and to quantify the emissions from these scrubbers. The two hypergolic fuels under investigation were monomethyl hydrazine (MMH fuel) and nitrogen tetraoxide (N_2O_4 oxidizer). Tests were conducted during the month of August 1980. This report presents the results of the testing of the hypergolic fuel vapor scrubbers.

PROCESS DESCRIPTION

Scrubber System

Two scrubbers were presented for testing, one each for MMH and N_2O_4. These scrubbers were manufactured by Martin-Marietta, Model Number S70-1095, and were designed to meet Kennedy Space Center Specification No. 79K08492. Each scrubber is a portable, skid-mounted system roughly 6 by 6 by 10 feet high. The system contains four vertical-packed bed scrubbing towers. A 750-gallon scrubber liquor storage tank is located beneath the packed towers. Scrubbing liquor is pumped from the storage tank to spray heads located in the top of each tower. The liquid flows by gravity from each tower back into the storage tank. Gas flow through the towers is countercurrent to liquid flow. The gas flows through each of the towers in series. Figure 1 is a block diagram of the scrubber system.

The scrubber system is designed to handle a flow rate of 400 standard cubic feet per minute, much higher than was encountered during the testing. The scrubber inlet line is 6 inches in diameter. For this test the scrubber system was modified to accept a 3/4-inch gas inlet line. Sample taps and valves were provided by Boeing Services International (BSI) on the inlet and outlet lines of the scrubber and the scrubber liquor storage tank.

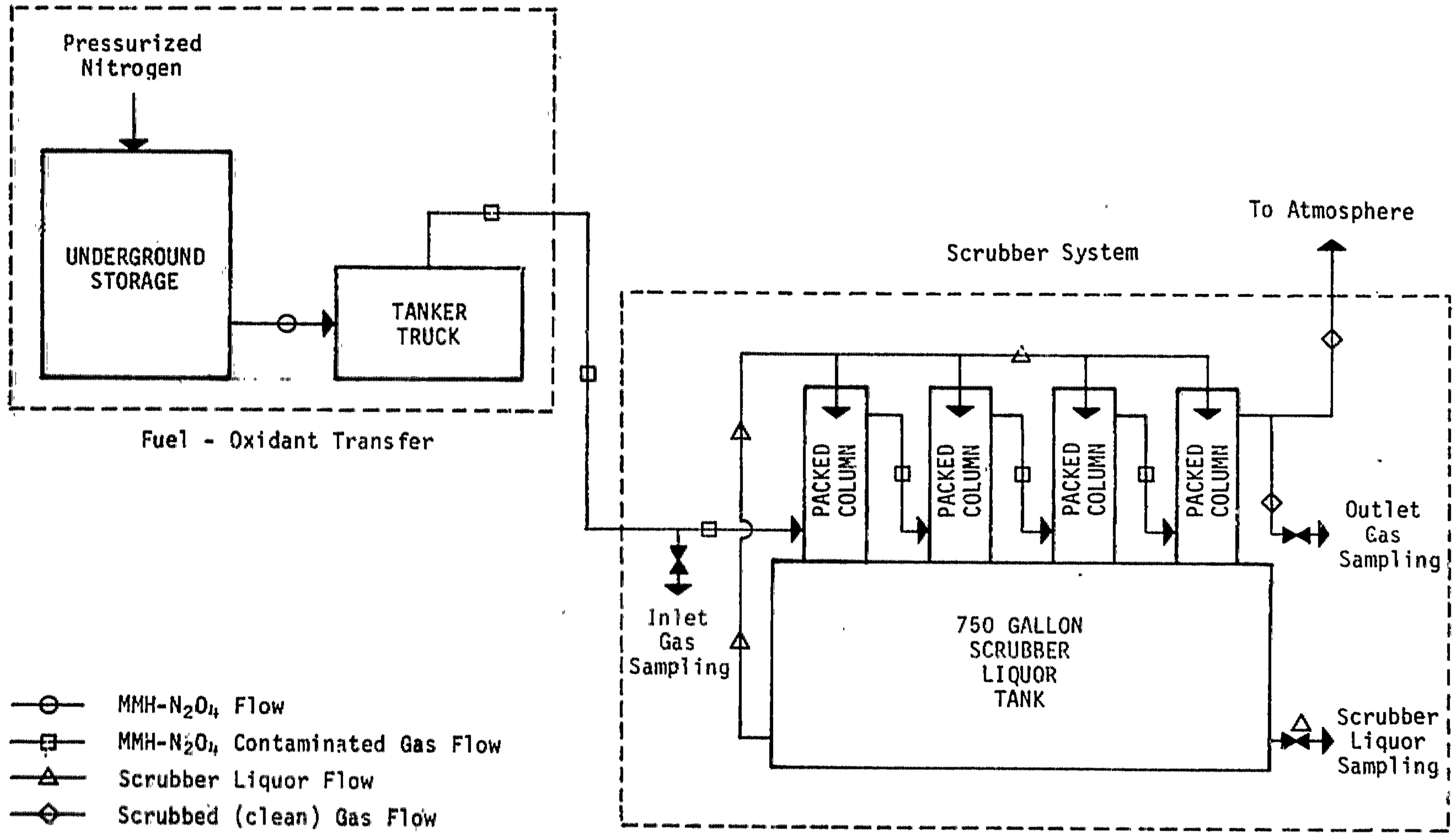

Figure 1. Block Diagram of Scrubber System Showing Sampling Locations (typical).

Scrubber Liquor

When scrubbing MMH vapors, the scrubbing liquor was a 14 percent solution of sodium citrate in water (citric acid). For removing N_2O_4 vapors, a water solution of 5 percent sodium hydroxide and 10 percent sodium sulfite was used.

Fuel/Oxidizer Transfers

Separate scrubber efficiency tests were conducted during fuel (MMH) and oxidizer (N_2O_4) transfer operations. The liquid propellants were transferred from a controlled storage tank to a tank truck. The transfer is a batch operation involving about 3,000 gallons per transfer. Pressurized gaseous nitrogen is used to force the propellant from one tank to the other. The tank being filled is vented through the scrubber to the atmosphere. The actual liquid transfer operation took about three quarters of an hour for MMH and about one and a quarter hours for N_2O_4. The propellant was then transferred back into the controlled storage tank, after chemical analyses showed that the propellant was not contaminated. Samples of the scrubber inlet and outlet gas streams, as well as scrubber solution samples, were collected during each transfer.

The transfer and scrubber operations were performed by BSI personnel. These operations incorporate very stringent safety regulations. All personnel within 50 feet of the transfer operation are required to wear special pressurized life support suits, called SCAPE suits. All personnel beyond the 50-foot limit must be upwind of the transfer site. No electrical equipment is allowed within 50 feet of the transfer site.

Prior to the start of a transfer, BSI personnel made all the piping hookups. Safety personnel then inspected all the connections. After this, safety personnel determined if the weather conditions were suitable for the transfer. (No transfers are allowed if there is an electrical storm in the area or if there is an inversion.) After the safety personnel gave their approval, the area was cleared and those working in the area put on their SCAPE suits. The transfer was then initiated. A total of 3 to 4 hours was required to complete the operations associated with a single transfer.

SECTION II

SUMMARY AND DISCUSSION OF RESULTS

Tables 1 and 2 present the results of testing conducted on the MMH and N_2O_4 scrubbers at the CCAFS. Table 1 lists the results for the MMH scrubber tests (Run Numbers MMH-R1 through MMH-R17). Table 2 lists the N_2O_4 scrubber test results (Run Numbers N_2O_4-R1 through N_2O_4-R10).

MMH SCRUBBER TEST RESULTS

Table 1 presents results for inlet and outlet MMH concentrations, scrubber solution MMH concentration and pH, and the gas flow rate to the scrubber. The inlet MMH concentration reported is the average of a series of grab samples, the number of samples dependent on the length of the run, collected during each run, as is the scrubber solution MMH concentration and pH. The inlet gas flow rate was calculated from the displacement effected by the transfer of liquid propellant per unit time. As part of the normal operations required during a fuel transfer, any excess pressure present in the receiving tank had to be vented through the scrubber prior to the initiation of the actual fuel transfer. This exhausted gas was not included in the air flow rate calculation since the initial tank venting operation was of short duration compared to the entire transfer time. However, gaseous sampling was conducted during this time.

The scrubber outlet MMH concentrations presented in Table 1 represent a variety of sampling conditions addressed in the footnotes to Table 1 and further explained in Section 4, Sampling Procedures. It became apparent to the test team shortly after the test program had started that measuring MMH at the low concentrations found in the scrubber outlet would be very difficult. Several variations in sampling procedures were tried. The method which proved most successful was the collection of a continuous outlet sample over the duration of four MMH transfers. This sample protocol was observed for runs MMH-R9-12, MMH-R13-16, MMH-R17, and MMH-R18. For reasons explained in Section IV, the contractor feels these results represent the most accurate MMH scrubber outlet concentrations measured during the sampling period. Values reported for the other runs should be considered approximate.

The average inlet MMH concentration for all the sampling runs was approximately 89,000 parts per million (ppm), and outlet MMH concentrations averaged 1.1 ppm (all values) and 2.3 ppm (results of four transfer samples, only). This represents a ollection efficiency for the scrubber of greater than 99 percent. Bear in mind, however, that observations made during collection of the inlet samples indicated the presence of droplets or a mist which could have

Table 1. SUMMARY OF MMH SCRUBBER SAMPLING

TEST NO.	INLET GAS FLOW (ACFM)	INLET MMH CONCENTRATION (PPM)	OUTLET MMH CONCENTRATION (PPM)	SCRUBBER SOLUTION MMH CONCENTRATION (G/L)	SCRUBBER SOLUTION pH
MMH-R1	11.8	56,200	~0.5[1]	0.4	1.59
MMH-R2	11.0	191,000	~0.3[1]	1.1	1.84
MMH-R3	13.7	201,000	~0.3[1,2]	2.2	1.98
MMH-R4	11.6	82,000	~0.6[3]	3.8	2.09
MMH-R5	15.5	83,200	~0.5[3]	4.1	2.26
MMH-R6	10.6	60,300	~0.5[4]	6.2	2.38
MMH-R7	13.1	53,500	~0.2[4]	6.9	2.46
MMH-R9	12.2	188,000	0.4[5]	7.6	2.57
MMH-R10	13.6	93,800	0.4[5]	7.2	2.61
MMH-R11	10.3	42,700	0.5[5]	8.6	2.74
MMH-R12	13.4	38,200	0.6[5]	8.7	2.82
(MMH-R9-R12)	(12.4)	(90,700)	(1.6[6])		
MMH-R13	11.6	24,200	0.5[7]	10.1	2.89
MMH-R14	12.4	71,900	0.4[7]	11.1	2.93

1 Average of short term samples - high sampling rate prevented good sample retention.

2 R3 CT2 (statistically rejected via 4D Method) not employed because of inordinately high values, probable sample contamination.

3 Runs R4 and R5 were comparative runs, comparing collection efficiency of H_2SO_4, citric acid, and HCl; H_2SO_4 results are reported.

4 Starting with run R6, extended impinger train to six impingers, each analyzed separately; results reported are for the sum of all impingers in a train. Also each of these continuous trains ran at different sampling rates to investigate effects on sample retention.

5 These trains ran as an adjunct to 4 transfer samples (see footnote 6).

6 These values represent an outlet sample collected over the last four runs (R9-R12, R13-R16) at reduced flow rate. Also, 6 impingers used for train; each impinger analyzed separately. These samples, in contractor's opinion, represent most accurate analysis of scrubber outlet concentration.

7 Dual impinger train, ran as an adjunct to 4 transfer samples referred to in footnote 6.

8 As in Runs R9-R12 and R13-R16, these samples were collected over a total of four transfers. Different values represent results from 3 separate trains, each sampled at different sampling rates. These values are considered more accurate results as in footnote 6.

9 Fresh scrubber solution.

Table 1. SUMMARY OF MMH SCRUBBER SAMPLING--Concluded

TEST NO.	INLET GAS FLOW (ACFM)	INLET MMH CONCENTRATION (PPM)	OUTLET MMH CONCENTRATION (PPM)	SCRUBBER SOLUTION MMH CONCENTRATION (G/L)	SCRUBBER SOLUTION pH
MMH-R15	11.1	45,000	0.8[7]	13.6	3.07
MMH-R16	14.3	57,400	0.6[7]	14.0	3.09
(MMH-R13-R16)	(12,4)	(49,600)	(1.9[6])		
MMH-R17	12.4	87,300	3.6[8] 4.3[8] 2.3[8]	16.1	3.23
MMH-R18	12.5	141,000	1.2[8] 1.6[8] 1.9[8]	3.1[9]	2.05[9]

1 Average of short term samples - high sampling rate prevented good sample retention.
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9 Fresh scrubber solution.

Table 2. SUMMARY OF N_2O_4 SCRUBBER SAMPLING

TEST NO.	INLET GAS FLOW (ACFM)	INLET NO_2 CONCENTRATION (PPM)	OUTLET NO_2 CONCENTRATION (PPM)	SCRUBBER SOLUTION SULFITE CONCENTRATION (%)	SCRUBBER SOLUTION pH
MON 3					
N_2O_4-R1	~7 [1]	578,000	56[2]	7.02	13.31
N_2O_4-R2	~6 [1]	106,000	43[2]	3.48	13.16
N_2O_4-R3	6.5	379,000	2,690[2,3]	2.33	12.83
N_2O_4-R4	6.6	271,000	68[2,4]	5.82	13.45[4]
N_2O_4-R5	7.2	660,000	687[2]	3.71	13.08
N_2O_4-R6	5.0	370,000	94[2,4]	5.12	13.47[4]
MON 1					
N_2O_4-R7	6.6	615,000	30[2]	4.25	13.17
N_2O_4-R8	6.9	339,000	2,120[2] 1,420[5]	0.90	11.94
N_2O_4-R9	2.0	514,000	6[2,4] 1[5]	5.63	13.61[4]
N_2O_4-R10	7.0	567,000	102[2] 58[5]	5.06	13.16
N_2O_4-R11	6.2	314,000	6,300[2,3]	1.90	11.68

1 Approximate values, trailer site glass not gradated.
2 Since retentive capacity of impinger trains was low, the average of the results from the short-term impinger train samples was used to generate this value.
3 Break through observed at the end of this run. (Visual indication of NO_2 gas exiting scrubber outlet.)
4 Replaced scrubber solution.
5 This value represents the NO concentration for the scrubber outlet (see Section IV).

been liquid MMH. The presence of the liquid MMH in the inlet sample would cause the concentration of gaseous MMH to appear very high. This would also cause the collection efficiency of the scrubber (on a gaseous basis) to be inordinately high.

Figure 2 represents a plot of scrubber solution MMH concentration and pH versus scrubber operating time. Predictably, the MMH concentration increased with operating time. Also predictably, considering that an acid-base reaction has been theorized by Kennedy Space Center personnel as the controlling reaction in the MMH scrubber, the scrubber solution pH also increased. After run MMH-R17, the scrubber solution was replaced with fresh citric acid. As can be seen in Table 1, the scrubber outlet concentration dropped considerably (from an average concentration for MMH-R17 of 3.4 ppm to 1.6 ppm for run MMH-R18), indicating that the scrubber solution may have been approaching exhaustion prior to its replacement.

N_2O_4 SCRUBBER TEST RESULTS

Table 2 presents the inlet and outlet nitrogen dioxide (NO_2) concentrations, the scrubber solution sulfite (SO_3) concentration and pH, and the inlet gas flow rate for tests conducted during a series of N_2O_4 transfers. As for the MMH transfers, the inlet gas flow rate was calculated from the displacement effected by the liquid N_2O_4 transfer. Inlet NO_2 concentrations and scrubber solution values are the average of several grab samples collected during each transfer.

As explained in the footnotes to Table 2 and further explained in Section IV - Sampling Procedures, only those outlet NO_2 samples collected over intervals of an N_2O_4 transfer were used to determine the scrubber outlet NO_2 concentration for that transfer (i.e., scrubber outlet NO_2 samples collected over the duration of the transfer were not used in determining the outlet concentration).

As part of the sampling protocol, two blends of N_2O_4 were to be investigated: MON 1 (99 percent N_2O_4 and 1 percent NO) and MON 3 (97 percent N_2O_4 and 3 percent NO). Transfers NO_2-R1 through -R6 were conducted while MON 3 was being transferred. Runs NO_2-R7 through -R12 were conducted on MON 1.

As part of the sampling protocol, scrubber outlet samples were analyzed for nitric oxide (NO) during runs N_2O_4-R8, -R9, and -R10. These results are presented in Table 2 as the second outlet concentration value reported for these runs.

The inlet NO_2 concentration measured during the N_2O_4 transfer averaged 428,000 ppm. This value represents not only NO_2 gas, but also N_2O_4 droplets in the inlet gas stream, as droplets of N_2O_4 were observed during inlet sample collection (see Section IV, Sample Procedures). N_2O_4 reacts with the NO_2 absorbing solution as NO_2 does, thus giving the appearance of very high NO_2 concentrations in the inlet gas stream.

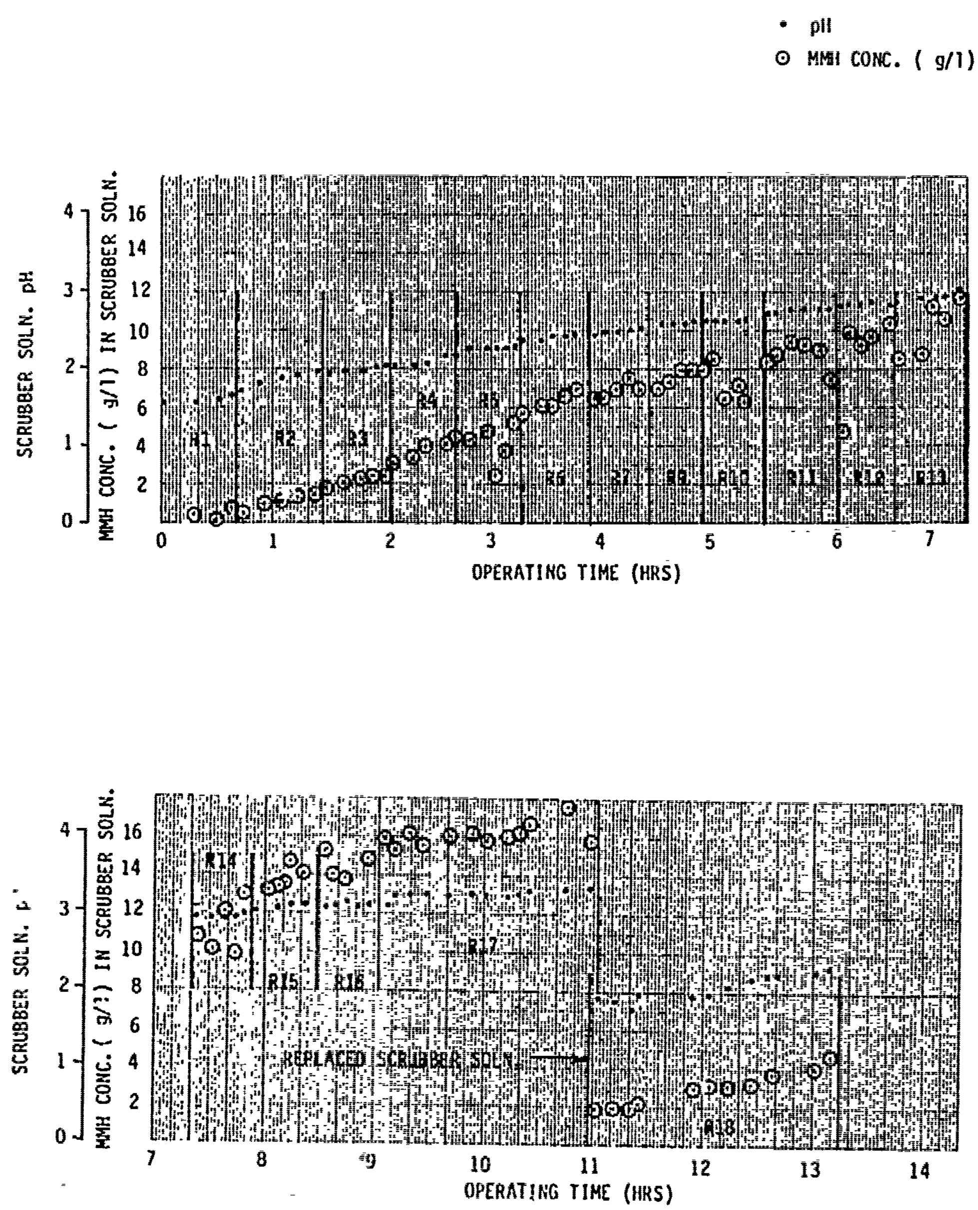
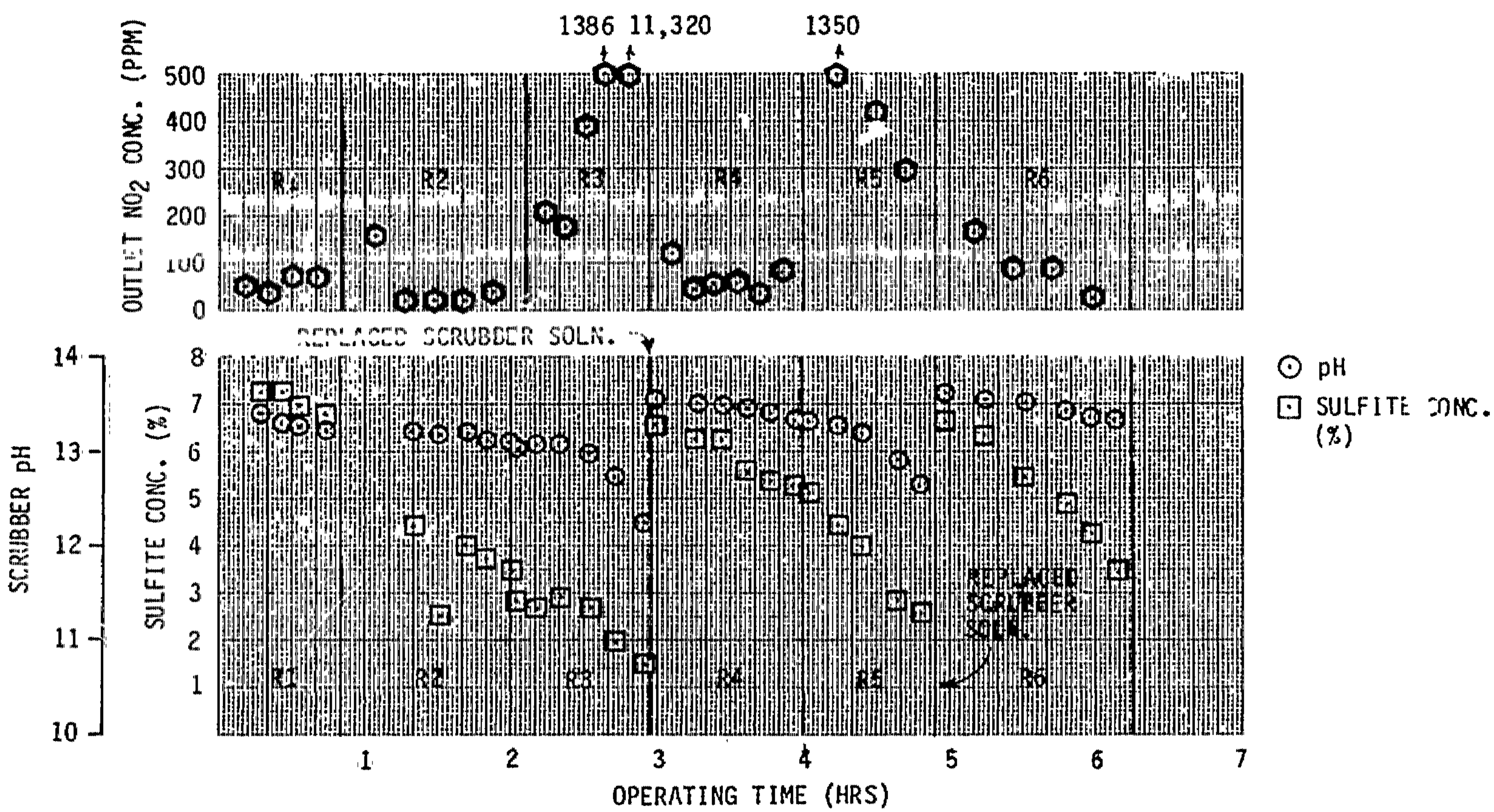


FIGURE 2. MMH Scrubber Operations, Scrubber Solution pH and MMH Concentrations versus Scrubber Operating Time

Figure 3a, 3b is a plot of scrubber liquor $SO_3^=$ concentration and pH, and scrubber outlet NO_2 concentrations versus scrubber operating time. The most obvious observation is the rapidity with which the scrubber solution was exhausted. (Each fresh charge of 10 percent $SO_3^=$ lasted for approximately 2.5 hours of scrubber operation.) At the completion of Runs N_2O_4-R3, -R5, -R8, and -R11, the scrubber solution appeared depleted, as as determined from three separate observations: the high NO_2 concentrations measured towards the end of these runs, the low $SO_3^=$ concentration measured in the scrubber solution at the same times, and the observation of a reddish-orange plume of gas (presumably NO_2) exiting the scrubber exhaust towards the end of Runs N_2O_4-R3 and -R11.

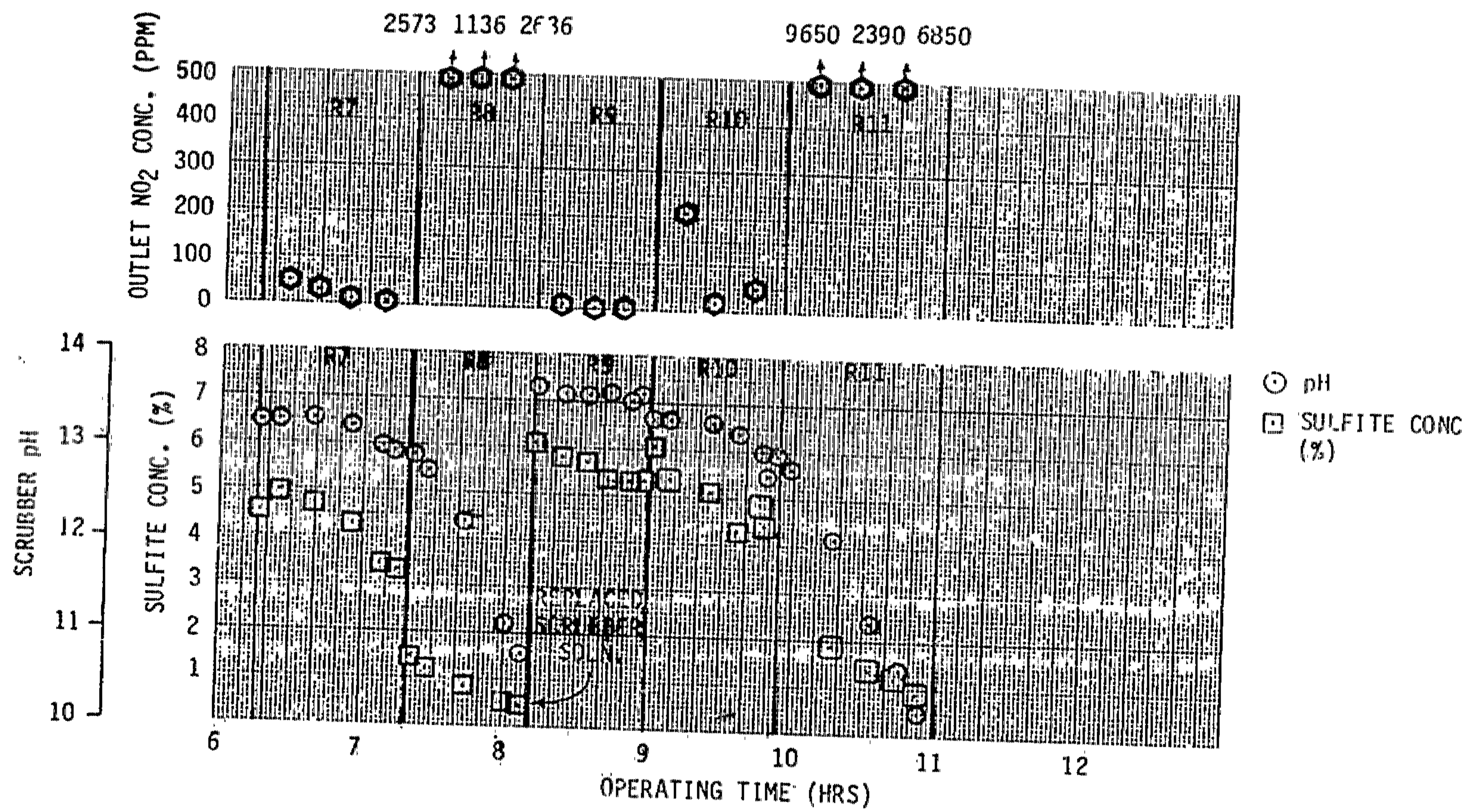
For Runs N_2O_4-R4 and -R6, it will be observed that outlet NO_2 concentration started high and dropped to lower values before the scrubber solution was depleted. No explanation can be given for this observation; however, it can be speculated that the scrubber system exhibits a lag effect due to the large scrubber volume in comparison to the gas flow rates.

Referring to Table 2, the outlet NO_2 concentration averaged 1,109 ppm for the 11 transfers conducted during this test program. If the results from those runs where the scrubber liquor $SO_3^=$ appeared exhausted are excluded the outlet NO_2 concentration averaged 57 ppm. Referring to Run N_2O_4-R9, the scrubber outlet concentration was only 6 ppm NO_2 during the run. This sample was collected during a special transfer in which the inlet gas flow rate was only 2.0 actual cubic feet per minute (ACFM) versus an average of 6.5 ACFM for the other 10 transfers. Since it can be assumed that the reaction of NO_2 gas with an aqueous $SO_3^=$ solution is a mass transfer limited reaction, the reduced gas flow rate may have enhanced the removal of NO_2 from the gas stream.



(a)

FIGURE 3. N_2O_4 Scrubber Operations, Scrubber Solution pH, Sulfite Concentration, Scrubber Outlet NO_2 Concentration versus Scrubber Operati'g Time



(b)

FIGURE 3. N_2O_4 Scrubber Operations, Scrubber Solution pH, Sulfite Concentration. Scrubber Outlet NO_2 Concentration versus Scrubber Operating Time (concluded)

SECTION III

SAMPLING EQUIPMENT

The equipment employed for sampling both the MMH and N_2O_4 gas scrubbers during this survey was identical.

INLET GASEOUS SAMPLES

For sampling the inlet gas, a 1/4-inch valved stainless steel tap with a rubber septum on the end was placed in the scrubber inlet line. Gaseous samples were extracted from this line using a 30-millimeter (ml) hypodermic syringe with a 2-inch Luer-Lok® needle. After the gaseous sample was collected and the sample absorbing solution (0.1 N HCl for MMH and Saltzman reagent for NO_2) was drawn into the syringe, the syringe was capped by setting a rubber cork on the end of the needle.

SCRUBBER LIQUOR SAMPLES

To collect scrubber liquor samples, a valved 1/4-inch stainless steel tap with a 1/4-inch sample line was placed in the discharge line of the scrubber solution recycle pump. Solution samples were drawn from this line into 500-ml borosilicte glass bottles with Teflon® lined caps. The 1/4-inch sample line extended to the bottom of the sample jar; efforts were made to ensure that the sample line was kept below the sample jar liquid level during sampling. This was done to minimize loss of MMH in the MMH scrubber solution and oxidation of $SO_3^=$ in the N_2O_4 scrubber solution during sample collection.

OUTLET GASEOUS SAMPLES

Figure 4 is a generalized diagram of the equipment employed to collect the outlet gaseous samples. A 1/4-inch stainless steel valved tap was placed in the outlet line of the scrubber. To this, the upstream manifold shown in Figure 4 was attached. To the manifold, a series of glass midget impinger trains containing the appropriate absorbing solution was coupled. The midget impingers were standard impingers fitted with glass bubbler tips rather than frits. All connecting tubing upstream of the glass impingers was either Teflon® or stainless steel.

Gas exiting the midget impinger trains passed through a silica gel drying tube to a polypropylene vacuum line of approximately 75 feet in length. This length was needed to ensure that all unprotected personnel and all electrical equipment was at least 50 feet

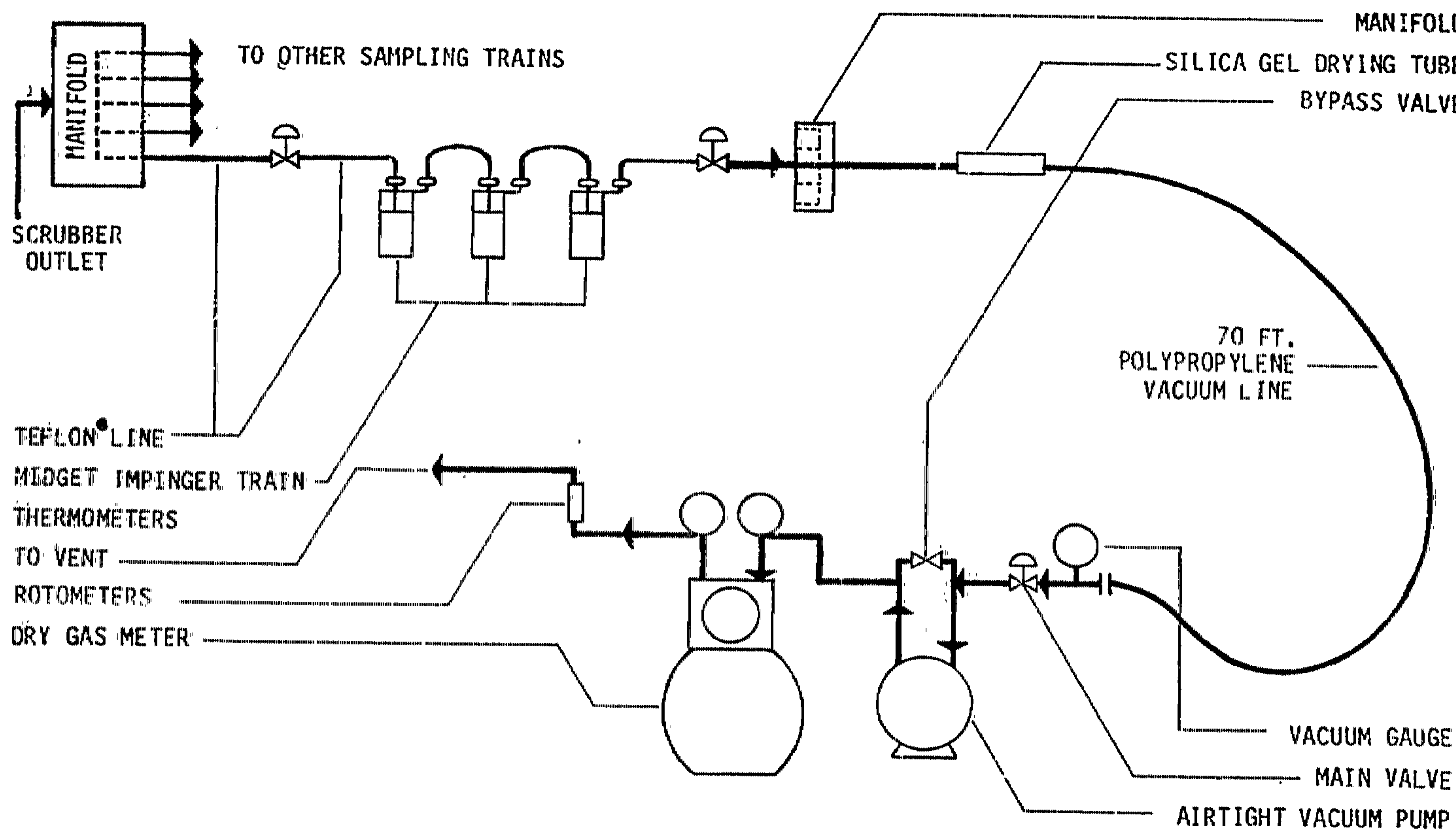


FIGURE 4. Typical MMH/NO_2 Scrubber Outlet Sampling Train

upwind from any potential MMH or N_2O_4 leaks, as required by safety regulations. Outlet gas samples were drawn through this assemblage using a RAC® (Research Appliance Corp.) vacuum pump - dry gas meter rotometer assembly for measuring sample flow rates and total gas volume sampled.

SECTION IV

SAMPLING PROCEDURES

This section addresses procedures employed for sampling the MMH and N_2O_4 scrubbers at the CCAFS. Procedures for sampling the inlet gas concentrations and scrubber liquors were basically the same for both MMH scrubber testing and N_2O_4 scrubber testing. Because of differences in the nature of the gases sampled, sampling for outlet gas concentrations varied considerably between MMH and N_2O_4 scrubbers. The first subsection of this section explains the procedures employed for sampling the inlet gas and scrubber liquors for both the MMH and N_2O_4 scrubbers. The second subsection describes sampling procedures for MMH scrubber outlet gas concentrations, and the third subsection addresses N_2O_4 scrubber outlet NO_2 gas concentration sampling.

INLET GAS AND SCRUBBER LIQUOR SAMPLING PROCEDURES

Inlet Gas Sampling Procedures

As originally planned, sampling for inlet gas concentrations was to have employed a midget impinger train assembly similar to the ones used for sampling the outlet gas. Prior to initiation of the test survey at the CCAFS, an investigation of the potential gas concentrations of MMH and NO_2 that might be encountered, based on vapor pressures, revealed that gas concentrations higher than could be effectively sampled with an impinger train would be encountered. Therefore, the decision was made to sample the inlet gas with 30-ml Luer-Lok® syringes.

Generally, the procedure for sampling inlet gas with the syringes was as follows. Within 5 minutes of the initiation of an MMH or N_2O_4 transfer, the first gas sample was extracted with a syringe. Immediately after this gas sample was collected, a known aliquot of absorbing solution was drawn into the syringe containing the gas sample. The syringe was then capped with a rubber cork and shaken to ensure good mixing between the gas and absorbing solution. Generally, for the MMH transfers, subsequent syringe samples were collected at approximately 10-minute intervals after the first sample collection. For N_2O_4 transfers, syringe samples were collected at 10- to 15-minute intervals after the first sample was collected. Gas sample volumes for MMH were were 15 ml and 10 ml for NO_2 samples. The absorbing solution for the MMH samples was 0.1 N HCl, and the amount of HCl drawn into the syringe after sample collection was 15 ml. For NO_2 samples, the absorbing solution was Saltzman Reagent, and the reagent aliquot was 20 ml.

After the completion of a transfer, the syringes containing gas sample and absorbing solution were transported to the field laboratory for sample recovery. Each syringe was vigorously shaken for about one minute, after which the liquid contents were expelled into a volumetric flask. Subsequently, the syringe was washed thoroughly with the appropriate absorbing solution; the wash contents were placed in the appropriate volumetric flask, and the flask contents were then taken up to volume with absorbing solution.

Scrubber Liquor Samples

The scrubber liquor samples were collected 1 to 2 minutes after the inlet gas samples. As explained in Section II, Sampling Equipment, borosilicate glass bottles were used to collect the scrubber solution samples. These samples were capped immediately after collection and were transported at the completion of a transfer to the field laboratory for analysis.

MMH OUTLET GAS SAMPLING PROCEDURES

At the inception of this test survey, the contractor's intent was to sample the outlet MMH concentration using midget impinger trains. The contractor proposed to take a total of six to eight outlet samples during each transfer. Two samples were to have been integrated samples collected throughout the entire transfer operation. The remaining four to six samples were to have been collected individually at 10- to 15-minute intervals during the transfer. These samples would have indicated if the concentration changed during the transfer.

At the completion of the first MMH transfer, results indicated that the outlet concentrations were very low, almost at the lower limit of detectability for the methodology employed to analyze the samples. Subsequently, for runs MMH-R2 and MMH-R3, the contents of each of the impingers was analyzed separately in an attempt to determine if the impinger trains were exhibiting good collection efficiency. Also, since the quantity of MMH being collected in the impinger trains was apparently so low, it was decided to increase the sampling time for each short-term impinger train. Although the results from runs R-2 and R-3 were not conclusive, indications were that two factors were effecting the low values observed for Run MMH-R1 outlet: poor retention of the subject MMH in the absorbing solution (0.1 N hydrochloric acid), and poor retention caused by high sampling flow rates which appeared to strip collected MMH from the lead impingers. Thus, the sampling team was confronted with the difficulty of collecting sufficient sample MMH for analysis in the time required to complete a transfer.

Runs MMH-R4 and -R5 were conducted to investigate different absorbing solutions: 0.1 N sulfuric acid (H_2SO_4), 0.1 N citric acid, and 0.1 N hydrochloric acid (HCl). The results of these runs indicated that the H_2SO_4 solutions were the best absorbing solutions. Subsequent sampling for MMH in the scrubber outlet

gas stream employed 0.1 N H_2SO_4 as the absorbing solution in the impinger trains. Also investigated during runs MMH-R4 and -R5 was the alteration of the impinger train from a total of three impingers to four impingers per train; this was done in an attempt to improve retention of MMH in the impinger train; however, the results were inconclusive.

Runs MMH-R6 and -R7 featured an expanded impinger train (six impingers) as well as variations in the sampling flow rates in order to investigate the effects flow rate had on collection efficiency. The results of these runs indicated that a lower flow rate effected better sample MMH retention, and the additional impingers were required to retain the MMH collected. Also, starting with these runs, no further short-term impinger train samples were collected.

Runs MMH-R9 through -R12, MMH-R13 through -R16, MMH-R17, and MMH-R18 represent scrubber outlet samples collected over the duration of four transfers. These samples were collected at a lower flow rate than attempted during any of the previous runs; since the sample flow rate was so low, it was necessary to collect a single sample over four transfers, thereby ensuring sufficient sample volume collection. These samples were analyzed by impinger (i.e., each impinger was analyzed individually); results indicated better sample MMH collection efficiency than had been previously observed. Because of this higher efficiency, the contractor believes that these outlet samples (Runs MMH-R12, -R16, -R17, and -R18) represent the most accurate values for scrubber outlet MMH concentrations. As an adjunct to the samples collected during these four transfers, runs MMH-R9 through -R16 featured two impinger trains of two impingers each, run parallel to the impinger train sample collected during the four transfers. These impinger trains were analyzed at the completion of each individual transfer. This was not done during runs MMH-R17 and -R18; instead, these two impinger trains were replaced with two impinger trains, each with six impingers per train, which were run for the duration of the four transfers. The results for each impinger train are reported individually as the outlet MMH concentration for runs MMH-R17 and -R18.

Table 3 presents a summary of sampling techniques, flow rates, etc., employed during the testing for MMH in the scrubber outlet gas stream.

Prior to the initiation of each sampling run, a series of impinger trains was charged with 20 ml of absorbing solution in each impinger at the field laboratory. The impinger trains were then transported to the transfer area and connected to the sampling manifold (Section III, Sampling Equipment) in preparation for sampling. After plumbing was accomplished, but before sampling, a leak check was performed using the following technique. The valve at the scrubber outlet sample line was closed and all intermediate valves were opened and the RAC® vacuum pump was turned on. The vacuum pump was allowed to run until 10 inches of vacuum was developed in

Table 3. OUTLET MMH SAMPLING SUMMARY

RUN NO.	SAMPLE TYPE[1]	SAMPLE FLOW RATE[2]	ABSORB. SOLUTION	NO. OF IMPINGERS	HOW[3] ANALYZED	COMMENTS[4]
R1-CT1	Continuous	1.85	HCl	3	Together	Very low MMH concentrations measured, at lower limits of detectability. T1-T5 reported.
CT2	Continuous	0.55	HCl	3	Together	
T1-T5	Short Term	0.92	HCl	3	Together	
R2-CT1	Continuous	0.87	HCl	3	Separately	Impingers analyzed independently as a check of impinger train collection efficiency. T4-T6 reported.
CT2	Continuous	0.68	HCl	3	Together	
T4-T6	Short Term	0.89	HCl	3	Separately	
R3-CT1	Continuous	1.77	HCl	4	Separately	Average of T1-T3 reported.
CT2	Continuous	0.73	HCl	3	Separately	
T1-T3	Short Term	0.81	HCl	3	Separately	
R4-CT1	Continuous	0.85	H_2SO_4	4	Separately	Check for absorbing solution applicability. CT1 reported.
CT2	Continuous	0.67	Citric Acid	4	Separately	
CT3	Continuous	0.82	HCl	4	Separately	
R5-CT1	Continuous	0.85	H_2SO_4	4	Separately	Check for absorbing solution applicability. CT1 reported.
CT2	Continuous	0.78	Citric Acid	4	Separately	
CT3	Continuous	0.85	HCl	4	Separately	

1 Sample type - Continuous - samples collected over the entire transfer.
Short Term - samples collected over a portion of a transfer; samples collected consecutively for duration of transfer.

2 Sample rate - standard liters per minute (68°F, 29.92 inches mercury).

3 How analyzed - contents of impingers analyzed independently or added together and analyzed as one.

4 Reported values refer to results shown on Table 1.

Table 3. OUTLET MMH SAMPLING SUMMARY--Continued

RUN NO.	SAMPLE TYPE[1]	SAMPLE FLOW RATE[2]	ABSORB. SOLUTION	NO. OF IMPINGERS	HOW[3] ANALYZED	COMMENTS[4]
R6-CT1	Continuous	0.84	H_2SO_4	6	Separately	Extended impinger train to check on collection efficiency. Also varied sampling flow rates. CT1 reported.
CT2	Continuous	0.65	H_2SO_4	6	Separately	
CT3	Continuous	0.47	H_2SO_4	6	Separately	
R7-CT1	Continuous	0.85	H_2SO_4	6	Separately	Continued investigation of varied sample flow rates. CT1, CT2, and CT3 results averaged and reported.
CT2	Continuous	0.64	H_2SO_4	6	Separately	
CT3	Continuous	0.50	H_2SO_4	6	Separately	
R8	---	---	---	---	---	Scratched because scrubber solution recycle pump was not started at the beginning of transfer.
R9-CT1	Continuous	0.82	H_2SO_4	2	Together	These samples were collected as an adjunct to four transfer samples collected. Average of CT1 and CT2 results reported for each run.
CT2	Continuous	0.66	H_2SO_4	2	Together	
R10-CT1	Continuous	0.82	H_2SO_4	2	Together	
CT2	Continuous	0.66	H_2SO_4	2	Together	
R11-CT1	Continuous	0.79	H_2SO_4	2	Together	
CT2	Continuous	0.68	H_2SO_4	2	Together	
R12-CT1	Continuous	0.87	H_2SO_4	2	Together	
CT2	Continuous	0.67	H_2SO_4	2	Together	

1 Sample type - Continuous - Samples collected over the entire transfer.
Short Term - Samples collected over a portion of a transfer; samples collected consecutively for duration of transfer.

2 Sample rate - standard liters per minute (68°F, 29.92 inches mercury).

3 How analyzed - contents of impingers analyzed independently or added together and analyzed as one.

4 Reported values refer to results shown on Table 1.

Table 3. OUTLET MMH SAMPLING SUMMARY--Continued

RUN NO.	SAMPLE TYPE[1]	FLOW RATE[2]	ABSORB. SOLUTION	NO. OF IMPINGERS	HOW[3] ANALYZED	COMMENTS[4]
R9 thru R12-CT3	Continuous	0.28	H_2SO_4	6	Separately	This sample was collected during R9 through R12 transfers at a greatly reduced sampling flow rate. This result reported independently.
R13-CT1	Continuous	0.79	H_2SO_4	2	Separately	These samples were collected as an adjunct to four transfer samples collected. Average of CT1 and CT2 results reported for each run.
CT2	Continuous	0.67	H_2SO_4	2	Separately	
R14-CT1	Continuous	1.74	H_2SO_4	2	Separately	
CT2	Continuous	0.62	H_2SO_4	2	Separately	
R15-CT1	Continuous	0.85	H_2SO_4	2	Separately	
CT2	Continuous	0.66	H_2SO_4	2	Separately	
R16-CT1	Continuous	0.83	H_2SO_4	2	Separately	
CT2	Continuous	0.63	H_2SO_4	2	Separately	
R13 thru R16-CT3	Continuous	0.28	H_2SO_4	6	Separately	This sample was collected as R9 through R12 sample. This result reported independently.

1 Sample type - Continuous - sample collected over the entire transfer.
Short Term - samples collected over a portion of a transfer; samples collected consecutively for duration of transfer.

2 Sample rate - standard liters per minute (68°F, 29.92 inches mercury).

3 How analyzed - contents of impingers analyzed independently or added together and analyzed as one.

4 Reported values refer to results shown on Table 1.

Table 3. OUTLET MMH SAMPLING SUMMARY--Concluded

RUN NO.	SAMPLE TYPE[1]	FLOW RATE[2]	ABSORB. SOLUTION	NO. OF IMPINGERS	HOW[3] ANALYZED	COMMENTS[4]
R17-CT1	Continuous	0.29	H_2SO_4	6	Separately	Run MMH R17 was collected during four transfers. Results of each impinger train reported (i.e., CT1, CT2, CT3)
CT2	Continuous	0.22	H_2SO_4	6	Separately	
CT3	Continuous	0.25	H_2SO_4	6	Separately	
R18-CT1	Continuous	0.30	H_2SO_4	6	Separately	Run MMH R18 collected and reported as Run MMH R17.
CT2	Continuous	0.23	H_2SO_4	6	Separately	
CT3	Continuous	0.20	H_2SO_4	6	Separately	

1 Sample type - Continuous - sample collected over the entire transfer.
Short Term - samples collected over a portion of a transfer; samples collected consecutively for duration of transfer.
2 Sample rate - standard liters per minute (68°F, 29.92 inches mercury).
3 How analyzed - contents of impingers analyzed independently or added together and analyzed as one.
4 Reported values refer to results shown on Table 1.

the impinger train, as measured via a vacuum gauge installed in the inlet manifold assembly. After the appropriate vacuum had been established, the impinger contents were checked for bubbling, and the dry gas meter register was observed for movement. If no bubbling was observed, and if the dry-gas meter registered less than 0.02 cubic feet in one minute, the system was considered leak tight. In no case was sampling allowed to proceed unless this leak check had been conducted satisfactorily. During sampling, the vacuum did not exceed 2 inches of vacuum.

At completion of a transfer, the impinger trains were disconnected from the sampling manifold, sealed with rubber caps, and transported to the field laboratory for sample recovery and analysis.

Sample recovery was accomplished by transferring the impinger contents into an appropriately sized volumetric flask. The impingers were then washed with the solution employed as the absorbing solution for that impinger train. The wash contents were then transferred to the volumetric flask, and the flask was brought up to volume with absorbing solution.

N_2O_4 OUTLET GAS SAMPLING PROCEDURES

As was the case with MMH sampling, the sampling team encountered difficulties in using impinger trains effectively to sample the scrubber outlet for the subject gas. For the N_2O_4 scrubber, the difficulty encountered resulted from the fairly high NO_2 gas concentration present in the scrubber outlet, and the poor transfer of NO_2 gas into the aqueous medium present in the impingers (Saltzman reagent).

Continuous impinger train samples were collected for the duration of this phase of the test program, as were short-term impinger trains. Not until after evaluation of the data at the completion of the test program, however, did it become obvious that the retention capacity of the continuous trains had been exceeded. For example, NO_2-R1 impinger trains CT1 and CT2 measured NO_2 concentrations in the scrubber outlet of 12 ppm and 28 ppm, respectively. Short term impinger trains T3 through T6 measured an average NO_2 concentration of 56 ppm for the same time period. This pattern repeated itself for continuous samples collected of the N_2O_4 scrubber outlet gas; for this reason, results reported for the N_2O_4 scrubber outlet NO_2 concentrations were calculated using the average of the short term impinger train results for each of the N_2O_4 transfers.

The techniques employed for setting up, leak checking, and recovering the impinger trains used to sample the N_2O_4 scrubber outlet were identical to those used for the MMH scrubber. (See previous heading, MMH Outlet Gas Sampling Procedures.)

For Run NO_2-R1, each impinger was filled with 20 ml of Saltzman reagent; however, due to the high NO_2 concentrations measured during

this run, the decision was made to increase the amount of Saltzman reagent to 30 ml in each impinger. After analysis of NO_2-R4 had been completed, results indicated that three impingers per train, as originally planned, were not sufficient. The decision was made to increase the number of impingers per train to six for run NO_2-R5 and all subsequent N_2O_4 transfers. However, even with six impingers in series, small amounts of NO_2 were observed in the last impinger of all trains. (Saltzman reagent turns pink upon contact with low concentrations of NO_2.)

While the test program was underway, the desire was expressed for a quantification of the amount of nitric oxide (NO) being discharged from the N_2O_4 scrubber. Employed was a method from Air Pollution Sampling and Analysis, 2nd Edition, for sampling NO (Method 405 - Tentative Method of Analysis for Nitric Oxide Content of the Atmosphere), whereby an oxidation chamber is placed in a sampling train for converting NO to NO_2, and the NO_2 formed is measured using the standard Saltzman technique. This was done during runs NO_2-R8, -R9, and -R10, plumbing this NO sampling train at the end of an NO_2 sampling train (Train T6). The oxidation chamber was plumbed in after the NO_2 impinger train, and the NO train followed the oxidation chamber.

Table 4 presents a summary of sampling techniques, flow rates, etc., employed during the testing for NO_2 in the scrubber outlet gas stream.

Table 4. OUTLET N_2O_4 SAMPLING SUMMARY

RUN NO.	SAMPLE TYPE[1]	SAMPLE FLOW RATE[2]	ABSORB. SOLUTION	NO. OF IMPINGERS	HOW[3] ANALYZED	COMMENTS[4]
R1-CT1	Continuous	0.49	20 ml Saltzman	3	Together	Average of T3-T4
CT2	Continuous	0.22	20 ml Saltzman	3	Together	reported.
T3-T6	Short Term	0.29	20 ml Saltzman	3	Together	
R2-CT1	Continuous	0.23	30 ml Saltzman	3	Together	Average of T1-T5
CT2	Continuous	0.22	30 ml Saltzman	3	Together	reported
T1-T5	Short Term	0.30	30 ml Saltzman	3	Together	
R3-CT1	Continuous	0.22	30 ml Saltzman	3	Together	Average of T1-T5
CT2	Continuous	0.21	30 ml Saltzman	3	Together	reported
T1-T5	Short Term	0.32	30 ml Saltzman	3	Together	
R4-CT1	Continuous	0.22	30 ml Saltzman	3	Together	Average of T1-T6
CT2	Continuous	0.21	30 ml Saltzman	3	Together	reported
T1-T6	Short Term	0.26	30 ml Saltzman	3	Together	
R5-CT1	Continuous	0.23	30 ml Saltzman	6	Together	Average of T3-T5
T3-T5	Short Term	0.27	30 ml Saltzman	6	Together	reported
R6-CT2	Continuous	0.22	30 ml Saltzman	6	Together	Average of T3-T6
T3-T6	Short Term	0.25	30 ml Saltzman	6	Together	reported

1 Sample type - Continuous - samples collected over the entire transfer.
Short Term - samples collected over a portion of a transfer; samples collected consecutively for duration of transfer.

2 Sample rate - standard liters per minute (68°F, 29.92 inches mercury).

3 How analyzed - contents of impingers analyzed independently or added together and analyzed as one.

4 Reported values refer to results shown on Table 2.

Table 4. OUTLET N_2O_4 SAMPLING SUMMARY--Concluded

RUN NO.	SAMPLE TYPE[1]	SAMPLE FLOW RATE[2]	ABSORB. SOLUTION	NO. OF IMPINGERS	HOW[3] ANALYZED	COMMENTS[4]
R7-CT2	Continuous	0.21	30 ml Saltzman	6	Together	Average of T3-T6
T3-T6	Short Term	0.24	30 ml Saltzman	6	Together	reported
R8-CT2	Continuous	0.20	30 ml Saltzman	6	Together	Average of T4-T6
T4-T6	Short Term	0.25	30 ml Saltzman	6	Together	reported
T6	Short Term	0.25	30 ml Saltzman	2		NO sample
R9-CT2	Continuous	0.42	30 ml Saltzman	6	Together	Average of T4-T6
T4-T6	Short Term	0.24	30 ml Saltzman	6	Together	reported
T6	Short Term	0.24	30 ml Saltzman	2	Together	NO sample
R10-CT2	Continuous	0.21	30 ml Saltzman	6	Together	Average of T4-T6
T4-T6	Short Term	0.25	30 ml Saltzman	6	Together	reported
T6	Short Term	0.25	30 ml Saltzman	2		NO sample
R11-CT2	Continuous	0.20	30 ml Saltzman	6	Together	Average of T4-T6
T4-T6	Short Term	0.25	30 ml Saltzman	6	Together	reported

1 Sample type - Continuous - samples collected over the entire transfer.
Short Term - samples collected over a portion of a transfer; samples collected consecutively for duration of transfer.

2 Sample rate - standard liters per minute (68°F, 29.92 inches mercury).

3 How analyzed - contents of impingers analyzed independently or added together and analyzed as one.

4 Reported values refer to results shown on Table 2.

SECTION V

ANALYTICAL PROCEDURES

This section describes the analytical methods employed during the hypergolic vapor scrubber testing conducted at the CCAFS. The first subsection addresses the analytical techniques employed during MMH scrubber testing, while the second subsection addresses the N_2O_4 scrubber testing analytical methods. Test data summary tables, including example calculations, are presented in Appendix A.

MMH SCRUBBER ANALYTICAL TECHNIQUES

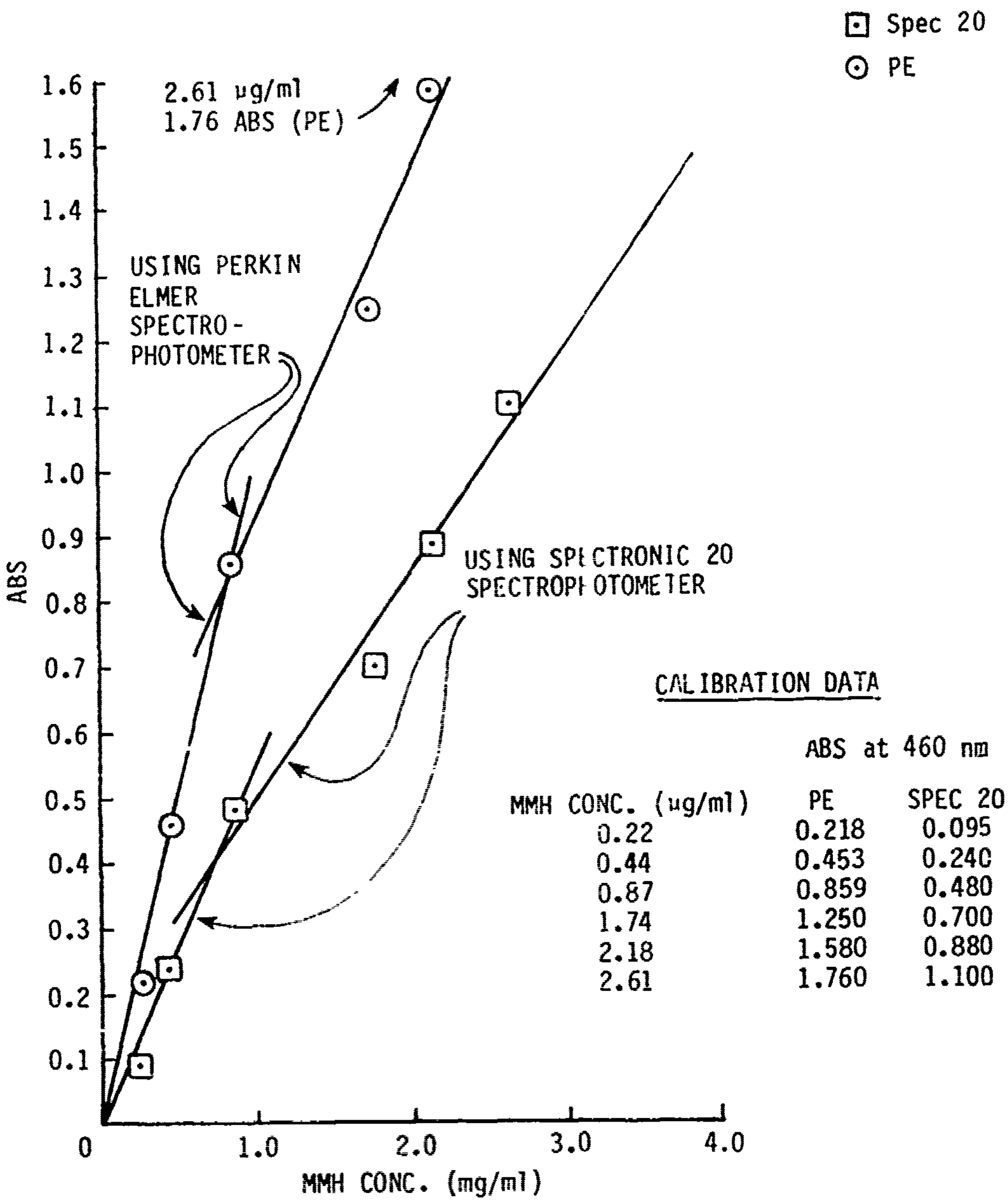
MMH Analysis

The analytical method for MMH determinations was the colormetric procedure whereby MMH reacts with p-dimethyl amino benzaldehyde (DAB) in an aqueous acid medium to form a highly colored product. Using a Perkin Elmer or Spectronic 20 spectrophotometer, with the instrument set to zero absorbence using a reagent blank, the absorbence was read at 460 namometers (nm) This absorbence was then compared with a calibration curve prepared using aqueous solutions of MMH and DAB.

Because of an equipment malfunction encountered during the test period involving the Perkin Elmer spectrophotometer, the field laboratory personnel were forced to use a Spectronic 20 single-beam spectrophotometer for a portion of the test period. Figures 5 and 6 show the calibration curves employed for MMH analysis using the two different spectrophotometers. Observe that these figures also address the use of DAB prepared: (1) in methanol, and (2) in water. Due to the shortage of methanol available at the field laboratory, it was necessary to analyze some of the MMH samples using DAB prepared in water instead of the specified methanol. The calibration curves drawn in Figures 5 and 6 were determined using linear regression.

pH Analysis

The MMH scrubber solution pH was measured using a standard glass pH electrode and an Orion pH meter. Samples were shaken well before measurement; samples were stirred with a magnetic stirring bar during measurement. The pH calibrations were done using standard buffer solutions.

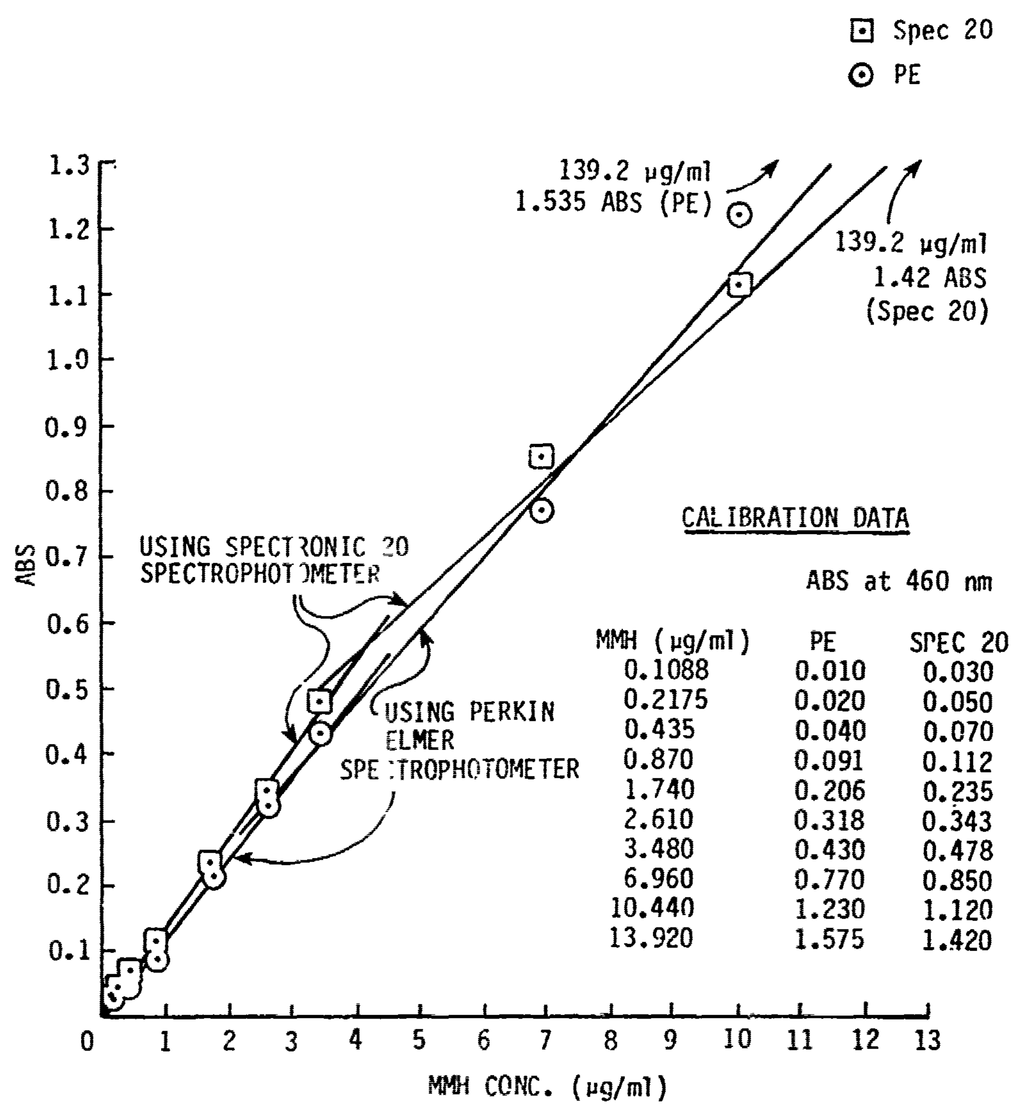


CALIBRATION DATA

	ABS at 460 nm	
MMH CONC. (μg/ml)	PE	SPEC 20
0.22	0.218	0.095
0.44	0.453	0.240
0.87	0.859	0.480
1.74	1.250	0.700
2.18	1.580	0.880
2.61	1.760	1.100

* p - Dimethylaminobenzeldehyde

FIGURE 5. Calibration Curve MMH Concentration versus Absorbance, DAB Prepared with Methanol



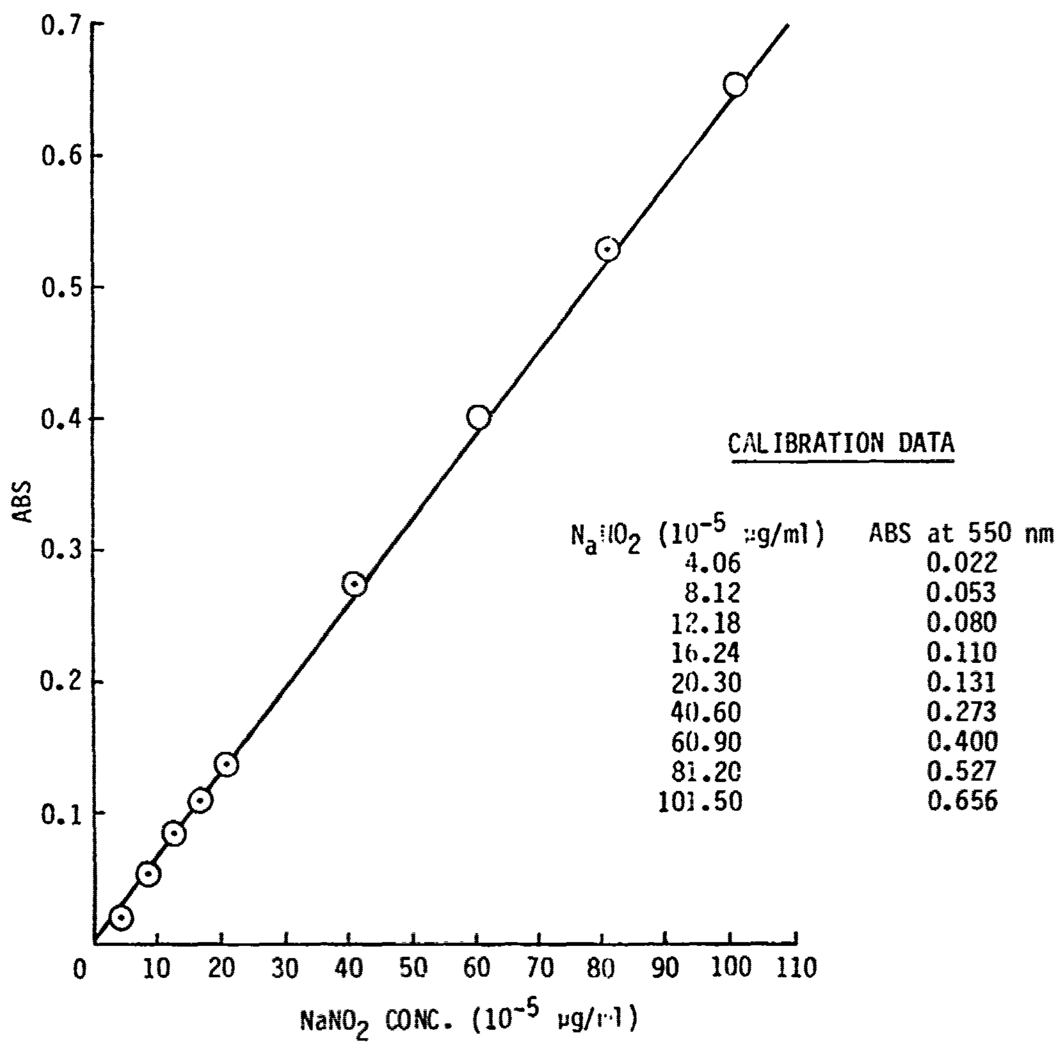
CALIBRATION DATA

ABS at 460 nm

MMH (µg/ml)	PE	SPEC 20
0.1088	0.010	0.030
0.2175	0.020	0.050
0.435	0.040	0.070
0.870	0.091	0.112
1.740	0.206	0.235
2.610	0.318	0.343
3.480	0.430	0.478
6.960	0.770	0.850
10.440	1.230	1.120
13.920	1.575	1.420

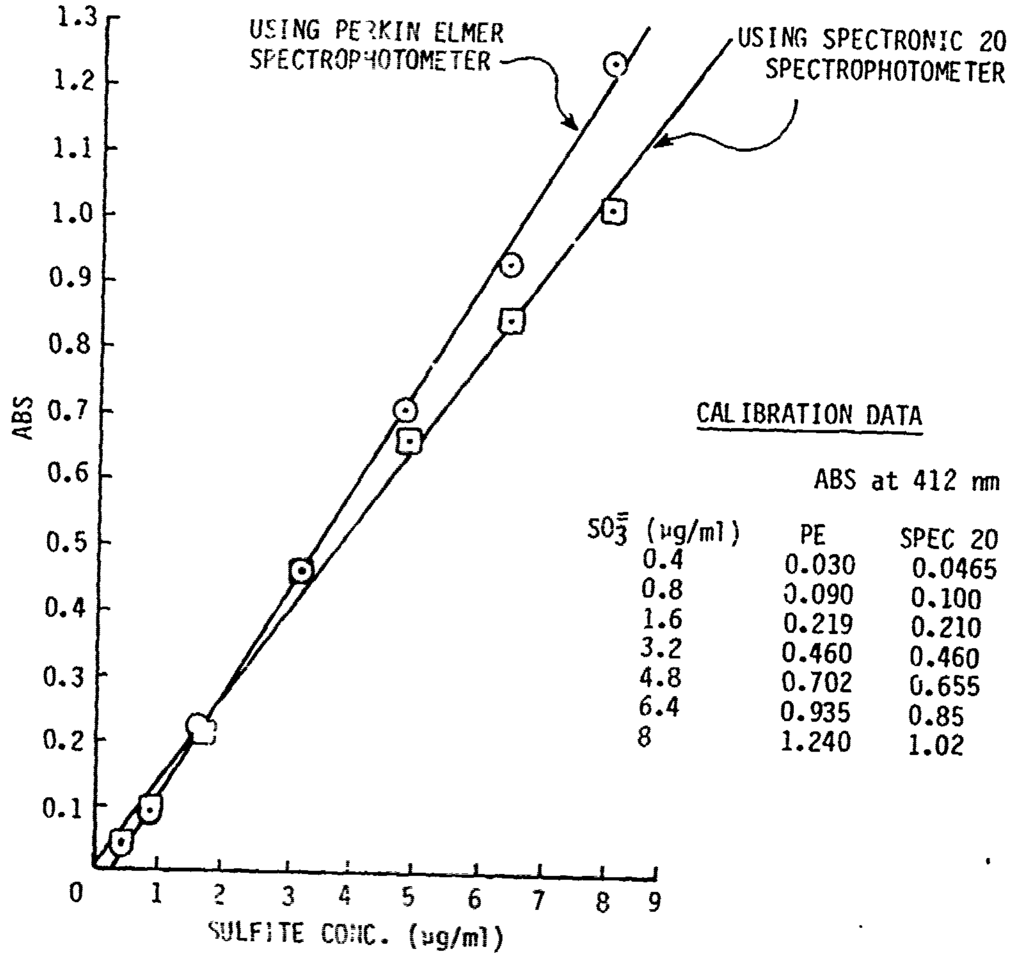
* p - Dimethylam nobenzeldehyde

FIGURE 6. Calibration Curve MMH Concentration versus Absorbance, DAB Prepared with Water



$NaNO_2$ (10^{-5} μg/ml)	ABS at 550 nm
4.06	0.022
8.12	0.053
12.18	0.080
16.24	0.110
20.30	0.131
40.60	0.273
60.90	0.400
81.20	0.527
101.50	0.656

FIGURE 7. Calibration Curve - $NaNO_2$ Concentration versus Absorbance (Perkin Elmer Spectrophotometer)



CALIBRATION DATA

	ABS at 412 nm	
$SO_3^{=}$ (µg/ml)	PE	SPEC 20
0.4	0.030	0.0465
0.8	0.090	0.100
1.6	0.219	0.210
3.2	0.460	0.460
4.8	0.702	0.655
6.4	0.935	0.85
8	1.240	1.02

FIGURE 8. Calibration Curve, Sulfite Concentration versus Absorbance

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APPENDIX A

TEST DATA SUMMARY TABLES
(INCLUDING EXAMPLE CALCULATIONS)

MMH/NO_2 GASEOUS EMISSIONS

Example Calculations

Sample No. N_2O_4 OUT-R10-24

OPERATIONAL DATA

a. Volume of dry gas sampled, meter conditions (V_m)	0.079 CF
b. Dry gas meter calibration coefficient (Y)	0.96
c. Average gas meter temperature (T_m)	561°R
d. Barometric pressure (P_b)	29.93 in. Hg
e. Sample absorbence (x)	0.049 ABS Units
f. Sample dilution factor (DF)	12,500
g. Slope of calibration curve (m)	648.833
h. Y-intercept of calibration curve (b)	1.4308 x 10^{-3}

CONSTANTS

a. Saltzman factor (empirical observation where one mole of NO_2 gas yields the absorbance of 0.72 moles of sodium nitrite) (K)	0.72
b. Conversion factor - liters per cubic foot (C_f)	28.3118 liters

DERIVED DATA

1. Conversion of dry gas sampled to standard liters

$$V_{mst} = (Y)(V_m)\left(\frac{T_{st}}{T_m}\right)\left(\frac{P_m}{P_{st}}\right)(C_f)$$

Where: T_{st} is the temperature at standard conditions (528°R) and P_{st} is the pressure at standard conditions (29.92 in. Hg).

$$V_{mst} = (0.96)(0.079)\left(\frac{528}{561}\right)\left(\frac{29.93}{29.92}\right)(28.3118) = 2.02 \text{ dry standard liters}$$

2. Calculation of milligrams of $NaNO_2$ collected in impinger train during sampling.

From the equation for a straight line, $y = mx + b$:

$$\text{mg } NaNO_2 = \left(\frac{X - b}{m}\right) DF$$

$$\text{mg } NaNO_2 = \left(\frac{0.049 - 1.4308 \times 10^{-3}}{48.333}\right) 12{,}500 = 0.9164 \text{ mg}$$

3. Conversion of milligrams of $NaNO_2$ to PPM of NO_2

$$\text{ppm} = \frac{\text{mg } NaNO_2}{V_{mst}} \; \frac{V_{stp}}{M} \; \frac{1}{K} \; \frac{1 \text{ g/mg}}{1{,}000} \; (1{,}000{,}000 \text{ parts/million})$$

Where V_{stp} is the volume in liters occupied by one mole at standard temperature and pressure (68°F and 29.92 in. Hg), and M is the molecular weight of $NaNO_2$ (69 g/mole).

$$\text{ppm} = \left(\frac{0.9164}{2.02}\right) \left(\frac{24.04}{69}\right) \left(\frac{1}{0.72}\right) 1000 = 219 \text{ ppm}$$

MMH/SULFITE CONCENTRATIONS
IN SCRUBBER LIQUORS

Example Calculations

Sample No. MMH-R1-SS1

OPERATIONAL DATA

a.	Sample absorbence (x)	0.337 ABS Units
b.	Sample dilution factor (DF)	500
c.	Slope of calibration curve (m)	0.98963
d.	Y-intercept of calibration curve (b)	3.9667×10^{-3}

DERIVED DATA

1. Calculate ug of MMH contained in 1 ml of scrubber liquor

$$\frac{ug}{ml}\,MMH = \left(\frac{x - b}{m}\right) DF$$

$$\frac{ug}{ml}\,MMH = \left(\frac{0.337 - 3.9667 \times 10^{-3}}{0.98963}\right) 500 = 168\ ug$$

2. Convert to grams MMH per liter

$$g/l\ MMH = \frac{ug/ml\ MMH}{10^{6}\ ug/g}\ (1000\ ml/l)$$

$$g/l\ MMH = \frac{168}{10^{6}}\,(1000) = 0.168\ g/l$$

MMH SCRUBBER SAMPLES

SAMPLE RUN TIME	GAS SAMPLE NUMBER	MMH CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	MMH CONC. (G/L)	pH
1050-1131 7/31/80					
1052	MMH-IN-R1-S1	23,300	MMH-R1-SS1	0.2	1.53
1107	S2	138,000	SS2	0.4	1.59
1117	S3	17,500	SS3	0.2	1.59
1127	S4	46,000	SS3	0.7	1.65
1102-1131.5	MMH-OUT-R1-CT1	0.1			
1104-1131.7	CT2	0.2			
1050-1100	T1	0.4			
1105-1115	T2	0.3			
1115-1125	T3	0.2			
1125-1127.8	T4	0.6			
1128-1131.7	T5	1.0			
0950-1037 8/1/80					
0951	MMH-IN-R2-S1	251,000	MMH-R2-SS1	0.6	1.71
1002.5	S2	242,000	SS2	1.0	1.82
1012.5	S3	166,000	SS3	1.1	1.85
1023.5	S4	210,000	SS4	1.4	1.90
1033.5	S5	88,000	SS5	1.5	1.94
0946-1039	MMH-OUT-R2-CT1-A	0.1			
	B	0			
	C	---			
0947-1039	CT2	0.2			
0947.5-1000	T4A	0.1			
	B	0.1			
	C	0.1			
1001-1021	MMH-OUT-R2-[illegible]-A	0.1			
	[illegible]	0			
	C	0.1			
1021-1038	T6-A	0.1			
	B	0.1			
	C	0.1			
1222-1258 8/1/80					
1224	MMH-IN-R3-S1	135,000	MMH-R3-SS1	1.8	1.92
1234.5	S2	90,000	SS2	2.0	1.96
1245	S3	47,900	SS3	2.3	1.97
1251	S4	131,000	SS4	2.4	2.00
1256.5	S5	600,000	SS5	2.4	2.04

MMH SCRUBBER SAMPLES (Continued)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	MMH CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	MMH CONC. (G/L)	pH
1221-1300	MMH-OUT-R3-CT1-A	0			
	B	0			
	C	--			
	C	0			
1222-1300	CT2-A	6.5			
	B	7.2			
	C	5.3			
1222-1231.5	T1-A	0.1			
	B	0.1			
	C	0.1			
1231.5-1247.5	T2-A	0.1			
	B	0.1			
	C	0.2			
1247.5-1300	MMH-OUT-R3-T3-A	0.1			
	B	0			
	C	0.1			
1226-1304.8					
8/4/80					
1228	MMH-IN-R4-S1	33,[illegible]00	MMH-R4-SS1	3.1	2.01
1236	S2	87,700	SS2	3.4	2.04
1242	S3	97,200	SS3	3.9	2.07
1253	S4	91,900	SS4	4.1	2.15
1258	S5	100,000	SS5	4.4	2.17
1224-1309	MMH-OUT-R4-CT1-A	0.2			
	B	0.1			
	C	0.2			
	D	0.1			
1225-1310	CT2-A	0.1			
	B	0.1			
	C	0.1			
	D	0.1			
1225-1310	CT3-A	0.1			
	B	0.1			
	C	0.1			
	D	0.1			
1351.5-1417					
8/4/80					
1353	MMH-IN-R5-S1	93,900	MMH-R5-SS1	4.3	2.24
1403	S2	89,700	SS2	4.8	2.25
1408	S3	82,300	SS3	2.4	2.26
1413	S4	86,500	SS4	3.7	2.27
1417.5	S5	63,700	SS5	5.2	2.27

MMH SCRUBBER SAMPLES (Continued)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	MMH CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	MMH CONC. (G/L)	pH
1350-1418	MMH-OUT-R5-CT1-A	0.1			
	B	0.2			
	C	0.1			
	D	0.1			
1351-1418	CT2-A	0			
	B	0			
	C	--			
	D	--			
1351-1418.5	CT3-A	0.2			
	B	0			
	C	0			
	D	0			
0926-1009 8/5/80					
0931	MMH-IN-R6-S1	31,800	MMH-R6-SS1	5.7	2.35
0941	S2	46,500	SS2	6.0	2.36
0947	S3	53,600	SS3	6.1	2.39
0954	S4	65,700	SS4	6.5	2.39
1003	S5	79,300	SS5	6.8	2.41
1010	S6	84,800			
0925-1012	MMH-OUT-R6-CT1-A	0.1			
	B	0.1			
	C	0.1			
	D	0.1			
	E	--			
	F	0.1			
0925-1012	CT2-A	0.2			
	B	0.1			
	C	0.1			
	D	0.1			
	E	0.1			
	F	0			
0926-1011.5	CT3-A	0.1			
	B	0.1			
	C	0			
	D	0.1			
	E	0			
	F	0			
1150-1222 8/5/80					
1155	MMH-IN-R7-S1	78,600	MMH-R7-SS1	6.5	2.42
1200	S2	53,300	SS2	6.5	2.43

MMH SCRUBBER SAMPLES (Continued)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	MMH CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	MMH CONC. (G/L)	pH
1207.5	MMH-IN-R7-S3	62,100	MMH-R7-SS3	6.9	2.46
1212.5	S4	24,500	SS4	7.4	2.48
1218.5	S5	63,500	SS5	7.0	2.50
1221.5	S6	38,800			
1150-1223	MMH-OUT-R7-CT1-A	0.1			
	B	0			
	C	0			
	D	0			
	E	0			
	F	0.1			
1150-1223	CT2-A	0.1			
	B	0.1			
	C	0.1			
	D	0			
	E	0			
	F	0			
1151-1223	CT3-A	0.1			
	B	0			
	C	0			
	D	0			
	E	0			
	F	0			
1120-1147					
8/6/80					
1128	MMH-IN-R9-S1	99,400	MMH-R9-SS1	7.0	2.54
1134	S2	121,000	SS2	7.4	2.57
1139	S3	65,800	SS3	7.9	2.57
1144	S4	466,000	SS4	7.9	2.59
--	S5	188,000			
1120.5-1148.7	MMH-OUT-R9-CT1-A	0.4			
	CT2-A	0.4			
1257-1328					
8/6/80					
1300.3	MMH-IN-R10-S1	49,400	MMH-R10-SS1	8.0	2.60
1304.5	S2	133,000	SS2	8.4	2.60
1310	S3	131,000	SS3	6.5	2.61
1316	S4	109,000	SS4	7.1	2.62
1320.5	S5	96,200	SS5	6.2	2.63
1328.8	S6	44,400			
1257-1330.3	MMH-OUT-R10-CT1	0.3			
1257.5-1330	CT2	0.4			

MMH SCRUBBER SAMPLES (Continued)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	MMH CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	MMH CONC. (G/L)	pH
0850-0929.9 8/7/80					
0854.5	MMH-IN-R11-S1	71,700	MMH-R11-SS1	8.3	2.70
0859.5	S2	25,900	SS2	8.7	2.71
0908	S3	22,900	SS3	9.3	2.74
0915.5	S4	29,900	SS4	9.1	2.76
0923	S5	68,900	SS5	8.9	2.76
0929.5	S6	37,000	SS6	7.5	2.76
0850-0933	MMH-OUT-R11-CT1	0.4			
0850.5-0933	CT2	0.6			
1055-1126.15 8/7/80					
1058.8	MMH-IN-R12-S1	17,000	MMH-R12-SS1	4.8	2.80
1103.8	S2	16,800	SS2	9.7	2.81
1109.5	S3	17,000	SS3	9.2	2.82
1115	S4	80,500	SS4	9.6	2.84
1121.3	S5	41,900	SS5	10.2	2.84
1125.3	S6	56,100			
1055-1127	MMH-OUT-R12-CT1	0.6			
1056-1127	CT2	0.6			
	CT3-A	0.5			
1122-1148.8	B	0.4			
1258-1330	C	0.3			
0851-0933	D	0.2			
1057-1127	E	0.1			
	F	0.1			
0844-0924.8 8/8/80					
0847	MMH-IN-R13-S1	18,900	MMH-R13-SS1	8.5	2.86
0859.5	S2	12,400	SS2	8.7	2.88
0907	S3	35,900	SS3	11.2	2.89
0913	S4	21,300	SS4	10.7	2.90
0921	S5	32,300	SS5	11.6	2.92
0844-0925	MMH-OUT-R13-CT1-A	0.2			
	B	0.2			
0844.5-0925	CT2-A	0.3			
	B	0.2			
1036.8-1109.8 8/8/80					
1040	MMH-IN-R14-S1	257,000	MMH-R14-SS1	10.7	2.92
1047	S2	25,700	SS2	10.1	2.92

MMH SCRUBBER SAMPLES (Continued)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	MMH CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	MMH CONC. (G/L)	pH
1053.7	MMH-IN-R14-S3	33,500	MMH-R14-SS3	12.1	2.92
1100.7	S4	19,900	SS4	9.8	2.93
1105.5	S5	23,200	SS5	12.9	2.96
1037-1110	MMH-OUT-R14-CT1-A	0.2			
	B	0.1			
1037.5-1100	CT2-A	0.3			
	CT2-B	0.1			
1005.8-1044.8 8/11/80					
1008.8	MMH-IN-R15-S1	94,500	MMH-R15-SS1	12.6	3.02
1013	S2	20,700	SS2	13.2	3.05
1020.3	S3	18,600	SS3	13.4	3.07
1026.5	S4	23,700	SS4	14.5	3.09
1034.3	S5	22,100	SS5	14.1	3.10
1040.5	S6	90,100			
1006-1045	MMH-OUT-R15-CT1-A	0.5			
	B	0.2			
1006.5-1045	CT2-A	0.5			
	B	0.3			
1206-1236.2 8/11/80					
1208.3	MMH-IN-R16-S1	97,000	MMH-R16-SS1	15.1	3.07
1212.3	S2	57,200	SS2	14.0	3.08
1219.5	S3	77,600	SS3	13.8	3.10
1226.7	S4	12,700	SS4	12.3	3.11
1233.7	S5	25,700	SS5	14.7	3.11
1236	S6	74,300			
1206-1237	MMH-OUT-R16-CT1-A	0.5			
	B	0.2			
1206.5-1237	CT2-A	0.3			
	B	0.2			
	MMH-OUT-R1C-CT3-A	0.6			
0845-0925	B	0.5			
1038-1110	C	0.3			
1007-1045	D	0.3			
1207-1237	E	0.1			
	F	0.1			
1039.5-1148 & 1311.5-1415.5 8/26/80					
1046	MMH-IN-R17-S1	1,170	MMH-R17-SS1	15.8	3.09
1101.5	S2	71,100	SS2	15.2	3.18
1110.7	S3	71,400	SS3	15.9	3.19

MMH SCRUBBER SAMPLES (Continued)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	MMH CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	MMH CONC. (G/L)	pH
1118.7	MMH-IN-R17-S4	80,600	MMH-R17-SS4	15.4	3.20
1132.7	S5	79,400	SS5	15.9	3.22
1145.5	S6	103,000	SS6	16.2	3.24
1316	S7	69,600	SS7	15.8	3.23
1328.7	S8	93,300	SS8	16.0	3.24
1334	S9	82,600	SS9	16.2	3.25
1340	S10	86,900	SS10	16.6	3.26
1401.8	S11	198,000	SS11	17.5	3.30
1415	S12	111,000	SS12	16.7	3.33
	MMH-OUT-R17-CT1-A	1.2			
	B	0.7			
1039.5-1148	C	0.5			
1311.5-1416.7	D	0.5			
	E	0.4			
	F	0.3			
	CT2-A	1.7			
	B	1.0			
1040-1148	C	0.6			
1312-1416.7	D	0.3			
	E	0.2			
1041-1148	T6-A	2.3			
1312.5-1418					
1055.5-1207.3 &					
1327.3-1431.5					
8/27/80					
1058	MMH-IN-R18-S1	598,000	MMH-R18-SS1	1.9	1.91
1108	S2	121,000	SS2	1.9	1.89
1117	S3	105,000	SS3	2.0	1.79
1121.8	S4	107,000	SS4	2.3	1.94
1152	S5	120,000	SS5	3.0	1.93
1201.7	S6	101,000	SS6	3.2	1.95
1331	S7	122,000	SS7	3.2	2.11
1344	S8	98,000	SS8	3.3	2.14
1352.5	S9	83,000	SS9	3.8	2.18
1355.9	S10	79,000	SS10	3.8	2.20
1419	S11	77,000	SS11	4.1	2.26
1427.3	S12	84,000	SS12	4.8	2.29
	MMH-OUT-R18-CT1-A	0.4			
	B	0.2			
1055.5-1207.5	C	0.2			
1327.5-1432.8	D	0.2			
	E	0.1			
	F	0.1			

MMH SCRUBBER SAMPLES--Concluded

SAMPLE RUN TIME	GAS SAMPLE NUMBER	MMH CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	MMH CONC. (G/L)	pH
	MMH-OUT-R18-CT2-A	0.5			
	B	0.4			
1056-1207.5	C	0.1			
1328-1433	D	0.2			
	E	0.2			
	F	0.2			
	MMH-OUT-R18-T6-A	0.6			
	B	0.5			
1056.5-1207.8	C	0.2			
1328.5-1433.2	D	0.2			
	E	0.2			
	F	0.2			

N_2O_4 SCRUBBER SAMPLES

SAMPLE RUN TIME	GAS SAMPLE NUMBER	NO_2 CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	$SO_3^=$ CONC. (%)	pH
1304-1355.6					
8/13/80					
1321	NO_2-IN-R1-S1	312,000	NO_2-R1-SS1	7.23	13.39
133[illegible]	S2	245,000	SS2	7.23	13.33
134[illegible]	S3	1,170,000	SS3	6.93	13.29
1349	S4	584,000	SS4	6.69	13.24
1304-1355	NO_2-OUT-R1-CT1	12			
1316-1355	CT2	28			
1316.5-1326.5	T6	47			
1326.5-1336.5	T5	34			
1336.5-1345.5	T4	70			
1345.5-1355.6	T3	72			
0907.5-1020.5					
8/14/80					
0937.5	NO_2-IN-R2-S1	104,000	NO_2-R2-SS1	4.43	13.23
0947.3	S2	144,000	SS2	2.52	13.21
0957.8	S3	118,000	SS3	3.97	13.18
1006.5	S4	130,000	SS4	3.71	13.15
1014.8	S5	131,000	SS5	3.45	13.12
1025	S6	11,400	SS6	2.78	13.09
0907.5-0959.5	NO_2-OUT-R2-CT1	56			
0932-1020	CT2	5			
0933-0950	T1	156			
0950-1000	T2	21			
1000-1010	T3	21			
1010-1018.8	T4	20			
1023-1029.5	T5	39			
1247.8-1333.3					
8/14/80					
1253	NO_2IN-R3-S1	82,000	NO_2 R3-SS1	2.68	13.09
1301.5	S2	191,000	SS2	2.86	13.07
1311.5	S3	405,000	SS3	2.66	12.99
1323	S4	550,000	SS4	1.97	12.75
1332.5	S5	664,000	SS5	1.47	12.25
1248-1333.3	NO_2-OUT-R3-CT1	373			
1248-1333.5	CT2	345			
1249-1259	T1	208			
1259-1309	T2	176			
1309-1319	T3	377			
1319-1329	T4	1,386			
1329-1333.8	T5	11,320			

N_2O_4 SCRUBBER SAMPLES (Continued)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	NO_2 CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	$SO_3^=$ CONC. (%)	pH
1303-1408 8/15/80					
1307.8	NO_2-IN-R4-S1	257,000	NO_2-R4-SS1	6.51	13.53
1326.3	S2	256,000	SS2	6.44	13.50
1335.1	S3	295,000	SS3	6.13	13.47
1344.8	S4	295,000	SS4	5.55	13.45
1354.7	S5	285,000	SS5	5.19	13.41
1405.3	S6	236,000	SS6	5.12	13.33
1303-1409	NO_2-OUT-R4-CT1	148			
1304-1409	CT2	64			
1305-1323	T1	120			
1323-1333	T2	48			
1333-1343	T3	61			
1343-1353	T4	59			
1353-1403	T5	37			
1403-1408	T6	85			
1248-1343.3 8/18/80					
1250.5	NO_2-IN-R5-S1	332,000	NO_2 R5-SS1	5.09	13.33
1303	S2	373,000	SS2	4.38	13.28
1314	S3	373,000	SS3	3.98	13.21
1328	S5	1,080,000	SS4	3.48	13.11
1337.5	S6	1,140,000	SS5	2.82	12.86
1248-1343.6	NO_2-OUT-R5-CT1	1,020	SS6	2.52	12.66
1249-1300.5	T3	1,350			
1300.5-1313	T4	421			
1313-1325.5	T5	292			
1056.5-1215 8/19/80					
1100	NO_2-IN-R6-S1	315,000	NO_2-R6-SS1	6.58	13.62
1116.5	S2	389,000	SS2	6.31	13.57
1132	S3	421,000	SS3	5.41	13.51
1151	S4	355,000	SS4	4.86	13.44
1053-1215	NO_2-OUT-R6-CT2	86	SS5	4.18	13.37
1057.5-1114.3	T6	170	SS6	3.39	13.33
1114.3-1129.3	T5	92			
1129.3-1149.3	T4	87			
1149.3-1215.3	T3	26			
1424.5-1525.5 8/19/80					
1427	NO_2-IN-R7-S1	6,710	NO_2-R7-SS1	4.59	13.27
1435	S2	28,600	SS2	4.98	13.30

N_2O_4 SCRUBBER SAMPLES (Continued)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	NO_2 CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	$SO_3^=$ CONC. (%)	pH
1449.5	NO_2-IN-R7-S3	87,300	NO_2-R7-SS3	4.73	13.29
1505.5	S4	1,640,000	SS4	4.33	13.20
1518.5	S5	1,320,000	SS5	3.45	13.02
1425-1525.7	NO_2-OUT-R7-CT2	11	SS6	3.39	12.92
1425.5-1432.5	T3	53			
1432.5-1447.5	T4	35			
1447-1502.5	T5	17			
1502.2-1525.8	T6	13			
<u>0921.5-1013.5</u>					
<u>8/20/80</u>					
0926	NO_2-IN-R8-S1	334,000	NO_2-R8-SS1	1.47	12.83
0931.5	S2	311,000	SS2	1.25	12.76
0948	S3	351,000	SS3	0.83	12.22
1006	S4	363,000	SS4	0.50	11.11
1011.8	S5	337,000	SS5	0.45	10.78
0920.5-1013.5	NO_2-OUT-R8-CT2	1,452			
0921-0929	T4	2,573			
0929-1004	T5	1,136			
1004-1014	T6	2,636			
1004-1014	(NO sample) T6	1,417			
<u>0922.3-1013</u>					
<u>8/22/80</u>					
0925.5	NO_2-IN-R9-S1	152,000	NO_2-R9-SS1	6.14	13.65
0938	S2	463,000	SS2	5.83	13.60
0948.3	S3	891,000	SS3	5.73	13.60
0957	S4	633,000	SS4	5.38	13.63
1007	S5	429,000	SS5	5.35	13.56
0922-1012	NO_2-OUT-R9-CT2	4	SS6	5.37	13.60
0923-0942	T4	12			
0942-0955	T5	6			
0955-1013	T6	1			
0955-1013	(NO sample) T6	1			
<u>1346.8-1439</u>					
<u>8/22/80</u>					
1350.8	NO_2-IN-R10-S1	313,000	NO_2-R10-SS1	6.08	13.36
1357.7	S2	280,000	SS2	5.41	13.35
1415	S3	441,000	SS3	5.17	13.29
1426.8	S4	742,000	SS4	4.32	13.20
1436.7	S5	1,060,000	SS5	4.91	13.00
1347-1439.5	NO_2-OUT-R10-CT2	76	SS6	4.48	12.75
1347.5-1355.9	T4	219			

N_2O_4 SCRUBBER SAMPLES (Concluded)

SAMPLE RUN TIME	GAS SAMPLE NUMBER	NO_2 CONC. (PPM)	SCRUBBER LIQUOR SAMPLE NUMBER	$SO_3^=$ CONC. (%)	pH
1355.9-1413.5	NO_2-OUT-R10-T5	63			
1413.5-1439.3	T6	24			
1413.5-1439.3	(NO) T6	58			
1325-1438					
8/25/80					
1329	NO_2-IN-R11-S1	332,000	NO_2-R11-SS1	3.13	12.98
1335	S2	354,000	SS2	2.87	12.85
1354	S3	327,000	SS3	1.89	12.12
1410.5	S4	231,000	SS4	1.40	11.20
1423	S5	389,000	SS5	1.21	10.69
1434	S6	252,000	SS6	0.92	10.25
1326-1438	NO_2-OUT-R11-CT2	3,860			
1326.5-1333	T4	9.650			
1333-1419	T5	2,390			
1419-1438	T6	6,850			